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Synthesis Characterization and Antioxidant Activity of Some Novel Pyrazolines

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Abstract

The present work deals with the synthesis of pyrazoline derivatives of halogen substituted chalcone with hydrazine hydrate in presence of NaOH in ethanol. The synthesized compound were structurally characterized on the basis of IR, ^{1}H-NMR and MASS spectral data. All of these newly synthesised compound were tested for their antioxidant activities using DPPH radical scavenging and OH radical scavenging methods.

Keywords: Pyrazoline, Antioxidant activity

1. Introduction

Pyrazoline and its derivatives represent one of the most active classes of heterocyclic compounds are vital biological agents. Pyrazoline is a small ring heterocycle containing two nitrogen have been under investigation for long time because of its important reported medicinal properties. They are as antitumour[1], antibacterial[2], antifungal[3], antibacterial[4], antiviral[5], anti-parasitic[6], antitubercular[7], insecticidal[8], anti-inflammatory[9], antidiabetic[10], antidiuretic[11], antianalgesic[12], antihelimentic[13], antihypolipaemic[14], antimalerial[15]. As a part of our programme aiming at the synthesis of different heterocyclic derivatives, we report here in the convenient synthesis of some new pyrazolines 2(a-j) from chalcone 1(a-j) treated with hydrazine hydrate in ethanol.

2. Method and Result

2.1 Chemistry

The chalcones[16] 1(a-j) were used for synthesis. The chemical hydrazine hydrate, ethanol and sodium hydroxide were obtained from SD Fine chemical local supplier and were used as such without further purification. The solvent were purified as per the standard procedure. Reflux method were used for synthesis of pyrazolines 2(a-j).

All synthesized compounds were characterized by spectral data (IR, ^{1}H-NMR, and MASS) which is consistent with the proposed structure.

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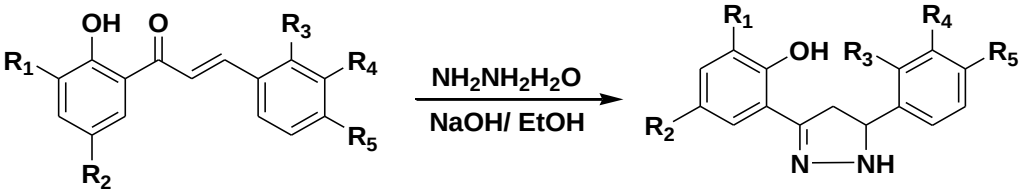

Substitution pattern and yield for compound 2(a-j)

Compound	R_1	R_2	R_3	R_4	R_5	M.P. ^{0}C	Yield%
1a	I	Cl	Br	H	H	152	60
2b	Br	Cl	Br	H	H	155	62
3c	Br	Cl	H	OCH_3	OH	148	65
4d	I	I	Br	H	H	172	58
5e	I	CH_3	Br	H	H	148	62
6f	Br	CH_3	Br	H	H	168	58
7g	I	I	H	OCH_3	OH	150	62
8h	I	CH_3	H	OCH_3	OH	145	65
9i	I	Cl	H	OCH_3	OH	140	65
10j	Br	CH_3	H	OCH_3	OH	145	68

3. Experimental Section

3.1 Chemistry

Melting point were determined in open capillary tube using melting point apparatous and are uncorrected. IR spectra were recorded on shimadzu FT-IR spectrophotometer using KBr pellets. ^{1}H-NMR spectra were measured in deuterated ($CDCl_3$) on an EA 400MHZ NMR spectrophotometer. Reaction was monitored by TLC using silica gel plate and pet ether, ethyl acetate (7:3) as a eluent system. The spot were visualized in an ultraviolet light at δλ= 254-266nm.

3.2 General Procedure for Synthesis of Pyrazolines

A mixture of chalcone (0.001 mol) hydrazine hydrate (0.002 mol) and catalytic amount of NaOH (0.250mg) in 15ml ethanol was refluxed for 3-4h. Reaction are monitored by TLC. After completion of reaction cooled product was filtered washed and recrystalized from ethanol to get pyrazolines 2(a-j).

1a: 2-(5-(2-bromophenyl)-4,5-dihydro-1*H*-pyrazol-3-yl)-4-chloro-6-iodophenol

IR(KBr, cm^{-1}): 1605(C=C), 3296(N-H); ^{1}H-NMR($CDCl_3$-d_6): δ 11.35(s, 1H, OH),7.99-7.31(m, 6H, Ar-H), 5.92(s,1H,NH), 4.66(d,1H,, CH of pyrazoline), 2.91(dd, 1H, CH_2 of pyrazoline), 3.06(dd, 1H, CH_2 of pyrazoline); Mass: (M^+): *m/z* 477.5

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1b: 2-bromo-6-(5-(2-bromophenyl)-4,5-dihydro-1H-pyrazol-3-yl)-4-chlorophenol
IR(KBr, cm^{-1}): 1606(C=C), 3342(N-H); ^{1}H-NMR($CDCl_3$-d_6): δ 11.50(s, 1H, OH),7.88-7.32(m, 6H, Ar-H), 5.59(s,1H,NH), 4.5(d,1H,, CH of pyrazoline), 3.52(dd, 1H, CH_2 of pyrazoline), 3.01(dd, 1H, CH_2 of pyrazoline); Mass: (M^+): *m/z* 430.
1c: 2-bromo-4-chloro-6-(4,5-dihydro-5-(4-hydroxy-3-methoxyphenyl)-1H-Pyrazol-3-yl)phenol
IR(KBr, cm^{-1}): 1600(C=C), 3302(N-H); ^{1}H-NMR($CDCl_3$-d_6): δ 11.50(s, 1H, OH), 12.87(s, 1H, OH),7.89-7.01(m, 6H, Ar-H), 6.04(s,1H,NH), 4.6(d,1H,, CH of pyrazoline),3.08(s,3H, OCH_3) 2.96(dd, 1H, CH_2 of pyrazoline), 3.11(dd, 1H, CH_2 of pyrazoline); Mass: (M^+): *m/z* 397.

3.4 Measurement of antioxidant activity

3.4.1 DPPH assay:

DPPH (2, 2, diphenyl-1-picrylhydrazyl) radical scavenging assay was carried out as per reported methods[17] with slight modification. Briefly, 1ml of test solution (Test compound) added to equal quantity of 0.1mM solution of DPPH in ethanol. After the absorbance at 517 nm. Ascorbic acid used as reference compound.

3.4.2 Hydroxyl radical scavenging assay

Hydroxyl radical scavenging activities were determined by the earlier reported method. The reaction cocktail contained 60 µl of 1 mM, $Fecl_3$, 90 µl of 1 mM 1,10-phenanthroline, 2.4 ml of 0.2 M phosphate buffer (pH 7.8), 150 µl of 0.17 M H_2O_2, and 1.5 ml of various concentration of individual compound. Reaction mixture kept at room temperature for 5 min incubation and absorbance was measured at 560 nm using spectrophotometer. α- Tocopherol was used a reference compound.

Table 2. Antioxidant activity

Sr. No.	Name of Compound	DPPH	OH
1	1a	15.91±0.89	8.80±0.76
2	1b	11.99±0.15	16.12±051
3	1c	17.99±0.51	28.25±0.1
4	1d	19.28±0.36	17.77±0.91
5	1e	12.29±0.45	12.55±0.78
6	1f	13.19±0.25	16.10±0.60
7	1g	14.19±0.15	24.58±0.66
8	1h	32.59±0.95	14.86±0.54
9	1i	11.19±0.55	15.14±0.43
10	1j	18.19±0.15	12.07 ±0.57
11	Standard	93.11±0.15	90.87±0.98

4. Conclusion

Here we reported some novel pyrazolines using substituted chalcones and hydrazine hydrate with better yield. These pyrazolines were characterized by their physical constant and spectroscopic data. *In-vitro* antioxidant activity of pyrazoline was studied by DPPH and OH radical scavenging activities. The compound showed antioxidant activity. It can be concluded that the +I effect of halogen always enhance the antioxidant activity as compared with the standard.

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